

# **2004 DOE Peer Review**

## **Stanford *In-Situ* High-Rate YBCO Process**

### **Transfer to Metal Tapes and Process Scale Up**

#### Presenters:

- Hong - Ying Zhai

*Stanford University*

- Jonathan Storer

*Los Alamos National Laboratory*

FY 2004 Funding: 100K

# Stanford Coated Conductor Program:

- **DOE – *Hammond and Zhai (since 4/1/04)***

- Characterize and Scale-Up Stanford High-Rate Deposition Process and Transfer to LANL
- Apply to Metal Tapes Provided by LANL

- **AFOSR Core Program – *Hammond, Geballe and Koster***

- Basic Materials Science of YBCO Relevant to Coated Conductor Deposition
- Exploration of YBCO 248 as an Alternative Coated Conductor Material
- Development and Application of FTIR as an In-Situ Diagnostic for YBCO Thin Film Deposition

- **AFOSR MURI – *Beasley and Moler***

- Development and Application of Scanning Probes for Coated Conductor Characterization – Large-Area, Sub-Micron Scanning Hall Probe and Scanning Tunneling Potentiometry



# Outline of Talk – by Task

- **Review of Stanford High-Rate Co-Evaporation Process**
- **Task I – Develop Robust Process for Scale-Up at LANL**  
(*Report by Jonathan Storer, LANL*)
  - **Result:** More practical approach to Cu atomic absorption rate control – dope Cu with RE. Rate control on RE. Avoids problem of strong atomic absorption of Cu.
- **Task II – Growth on LANL-IBAD Tapes**  
(*Report by Hong-Ying Zhai, Stanford*)
  - **Result:** Grew YBCO films on tape from 870° C – 940 ° C. Characterized by XRD and resistivity. Lowering the substrate temperature shows an improvement in film quality.
  - **Result:** Applied new FTIR characterization tool to tapes
    - Established directly temperature of film on tape during growth
    - Use FTIR reflectivity to monitor material state during deposition and processing.



# Review of Present Stanford High-Rate Process

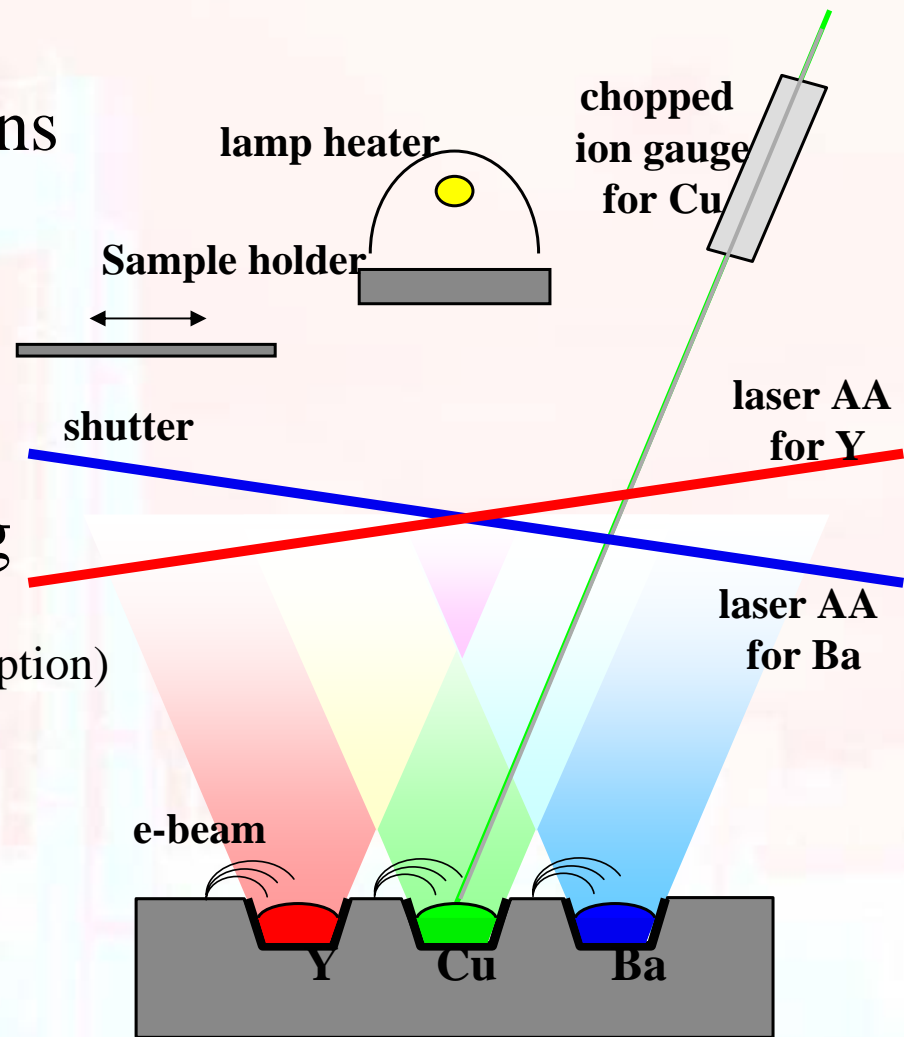
## ❖ Typical Deposition Conditions

- ▷ Pressure:  $10^{-5}$  Torr  $O_2$
- ▷ Deposition  $T_s \sim 900^\circ\text{C}$

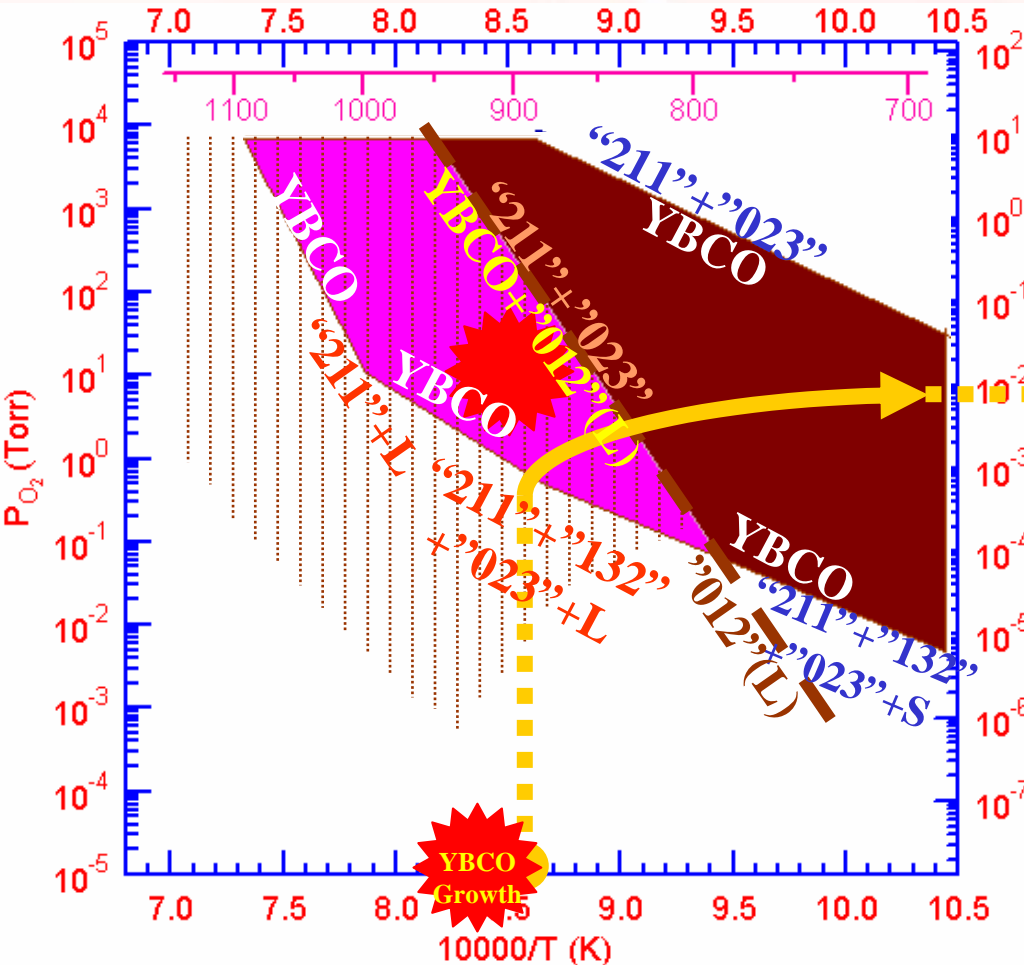
## ❖ Evaporation Rate Monitoring

- ▷ Y and Ba -- Laser AA (Atomic Absorption)
- ▷ Cu -- Chopped Ion Gauge (ERM)
- ▷ Cu absorption too strong to use AA\*

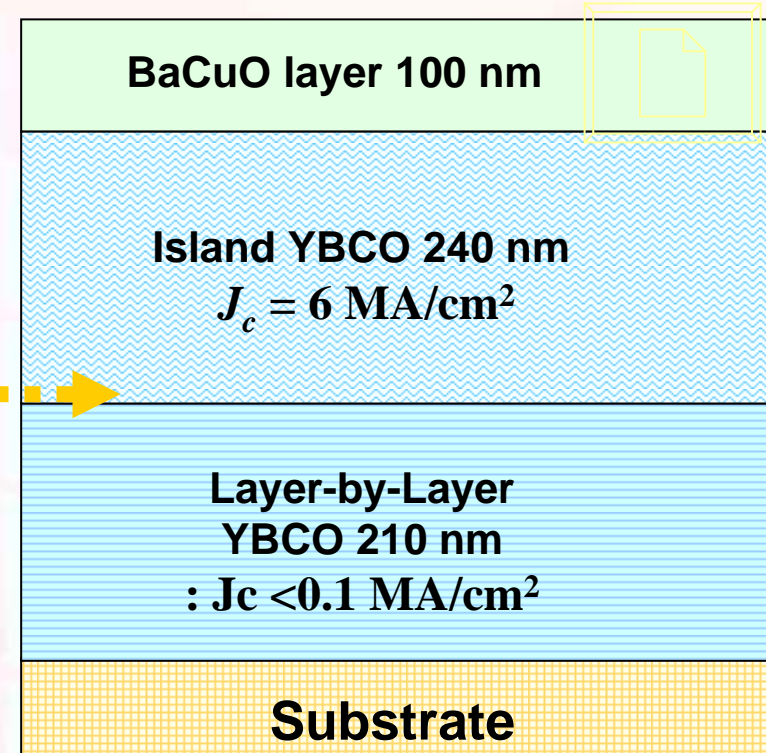
\* Storer (LANL) will discuss.



# Deposition Process & Resulting Film Profile



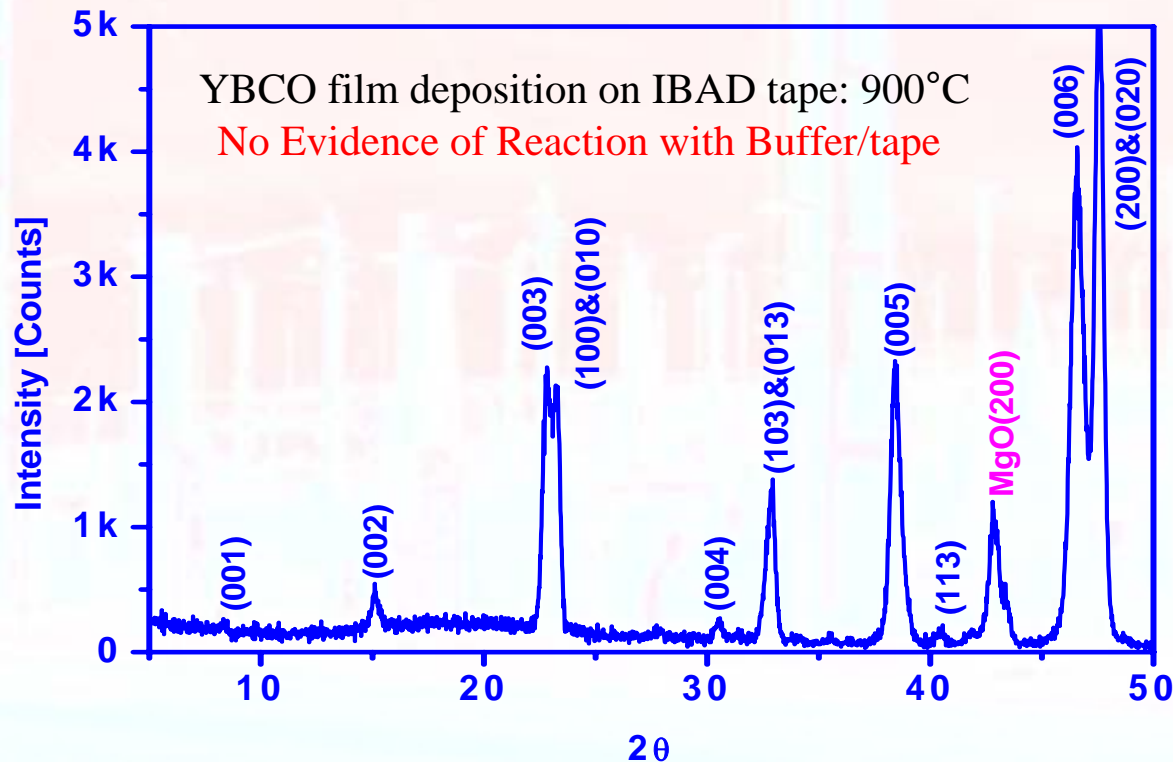
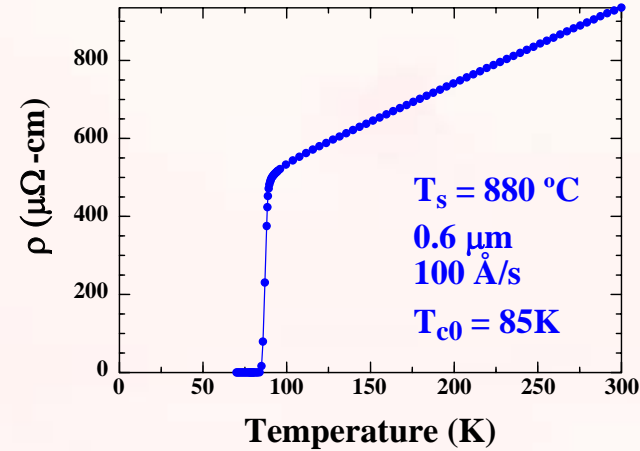
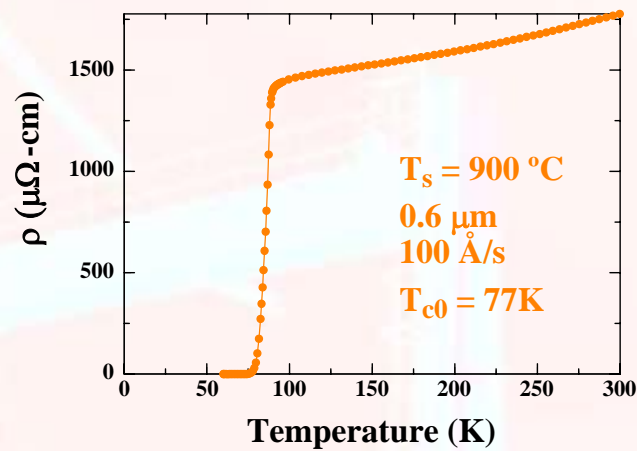
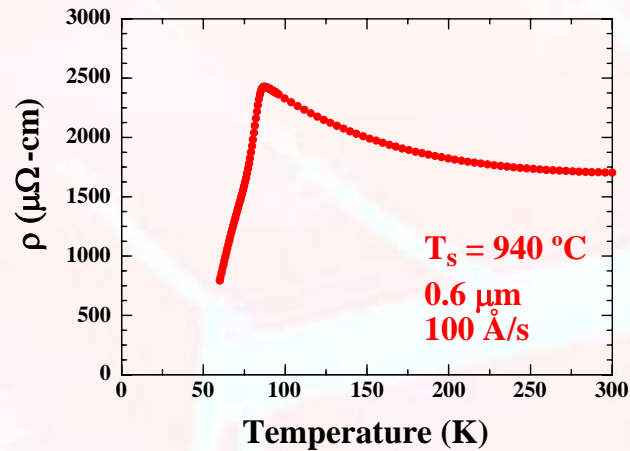
Stability Diagram



TEM & Depth profile show two layers of YBCO.  
(Note: This film was grown in one deposition)



# Results to Date on IBAD Tape



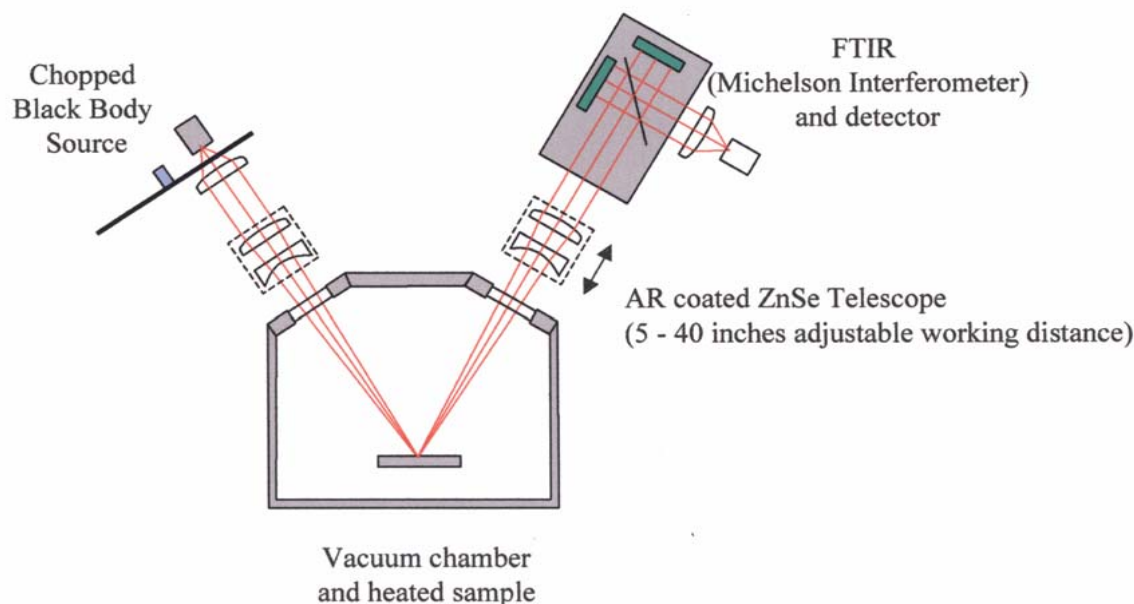
# Fourier Transform Infrared spectroscopy (FTIR)

Principle of Operation for Temperature Measurement

**M**easure radiance and reflectance as a function of  $\nu$  or wavelength:  $600\text{-}6000\text{ cm}^{-1} \sim 1.5\text{-}15\text{ }\mu\text{m}$

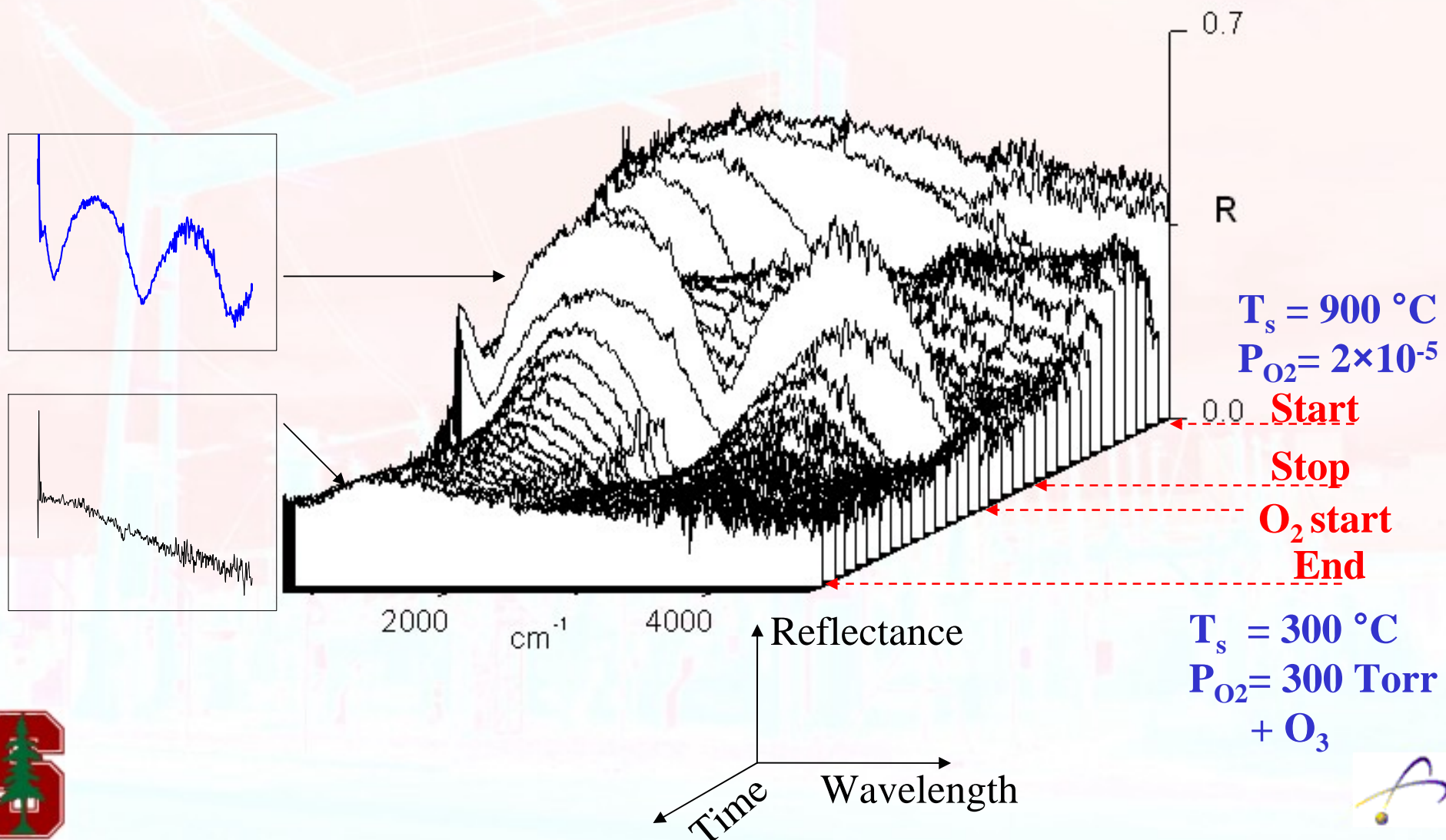
**R**eflectivity provides real time information of the thin film properties

**F**TIR is valuable asset to coated conductor process both deposition as well as post anneal steps





# FTIR *in situ* Observation of Film Growth





# *Ex situ* X-ray Heating cycle

Investigate the *ex situ* heating cycle on the phase transitions in  $\text{YBa}_2\text{Cu}_3\text{O}_7$  and Ba-Cu-O system on both IBAD tape and Single Crystal.

**Ba-Cu-O system:**

**Epitaxial  $\text{BaCuO}_2 + \text{CuO}$**

**Layer by Layer**

**Deposited by 2-target  
sequence by PLD**

**Ex situ X-ray system**

**Phillips Xpert**

**Dome Heat Stage (DHS)**

**280 mTorr  $\text{O}_2 + \text{Ar}$**

**Temperature:**

**100-900°  $\theta$ -2 $\theta$  scan / 25 °C**

**It was found** that the Ba-Cu-O epitaxial peak ( $\sim 29^\circ$ ) appears repeatedly with temperature sweeping: A possible indication of liquid-solid transition

$\text{YBa}_2\text{Cu}_3\text{O}_7$  system

Study the stability of heating cycle in different temperature and (0.28 & 760 Torr)  $\text{O}_2$  or  $\text{O}_2 + \text{Ar}$ . Found the information regarding to the phase transition of YBCO and information for co-evaporation or other low  $\text{O}_2$  deposition system.



# Summary

## 1. YBCO Deposited on IBAD Tape Substrate

- Lower deposition temperature is the direction to go.
- X-ray shows no reactions between YBCO/Buffer/Tape at 900°C.

## 2. We use Optical Infrared Reflectance (FTIR) to Monitor Phase Transitions in our Process – *in situ*

## 3. Use X-ray Dome Heater Stage to Study Structure Transition – *ex situ*.

# Next

Jonathan will give the presentation on the work in LANL

# Co-Evaporation of YBCO

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**Status of co-evaporation at LANL**

**Nd/Cu evaporation**

**Summary of LANL work**

**Opinion: Only reactive evaporation or a fast conversion process  
are commercial candidates.**

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**Electron beam evaporation has deposited films at 1  
 $\mu\text{m}/\text{sec}$  over an area of  $750\text{ cm}^2$  (15 cm x 50 cm)**

**An equivalent conversion process at  $0.01\text{ mm}/\text{sec}$  has  
an area of  $75000\text{ cm}^2$  (10cm x 75 m)**

## Co-evaporation system is operational at LANL

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**From 3M Company, formerly used for  $\text{BaF}_2$ -Y-Cu  
co-evaporation with limited *in situ* YBCO runs.**



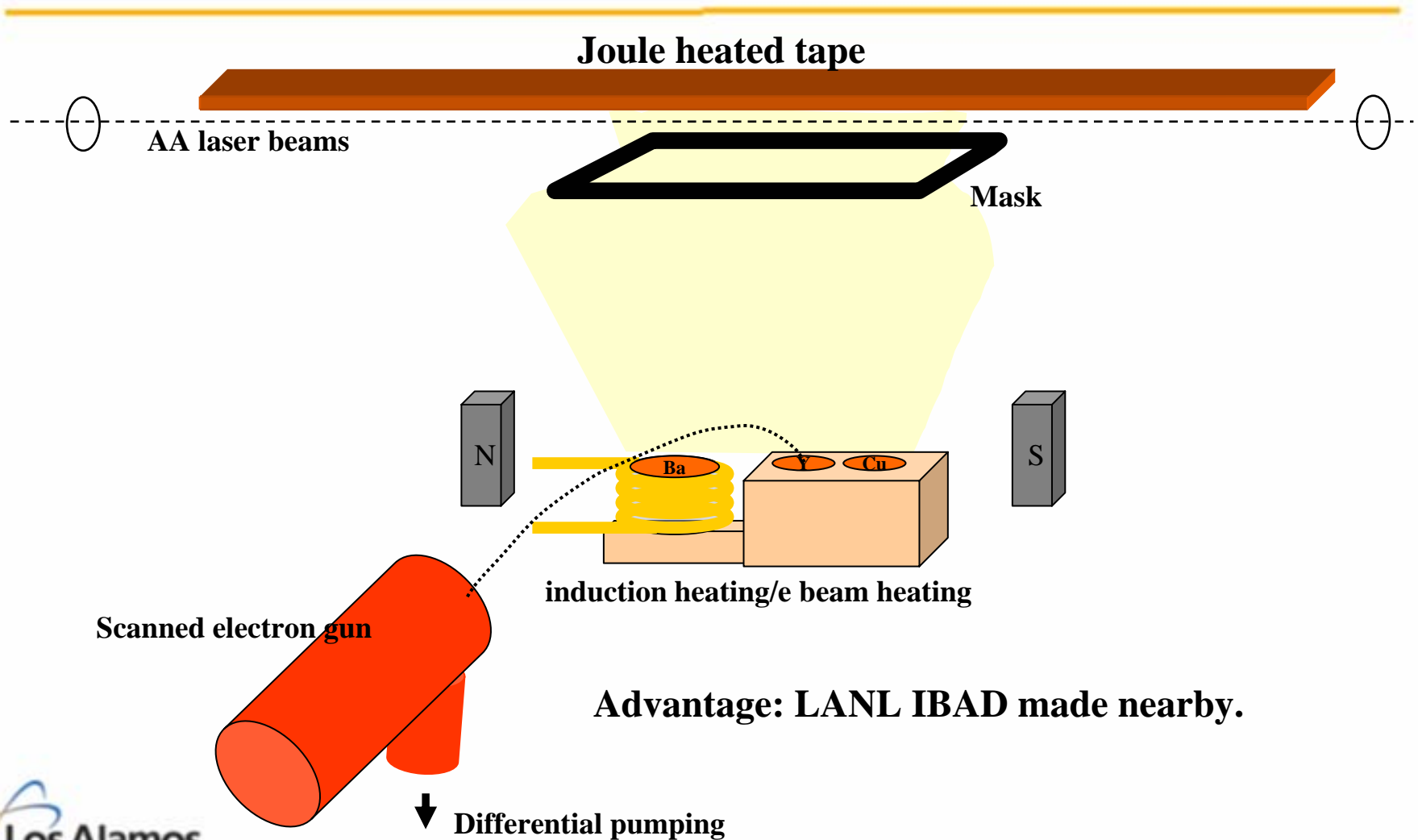
# Installation improved over previous



- Skipping beam, differentially pumped electron gun
- Tunable diode laser atomic absorption sensors
- 30 kW, 7.5 cm width.



# LANL Co-evaporation Schematic



**Advantage: LANL IBAD made nearby.**

# Atomic Absorption (AA) is feasible if

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1. A ground state or low lying metastable state line in the range of an available tunable diode laser.
2. Oscillator strength is favorable for absorption measurement.

# Picking the laser line

	Wavelength (nm vac)	Oscillator Strength	
Copper ground state	324.85	-0.062	Bad, Bad
	327.49	-0.359	Bad, Bad
Yttrium ground state	668.94	-2.000	Good, Good
	679.56	-1.820	Good, Good
Neodymium ground state	692.58	-1.530	Good, Good
	698.71	-1.940	Good, Good
	733.66	-2.140	Good, Good
Barium ground state	791.35	-2.000	Good, Good

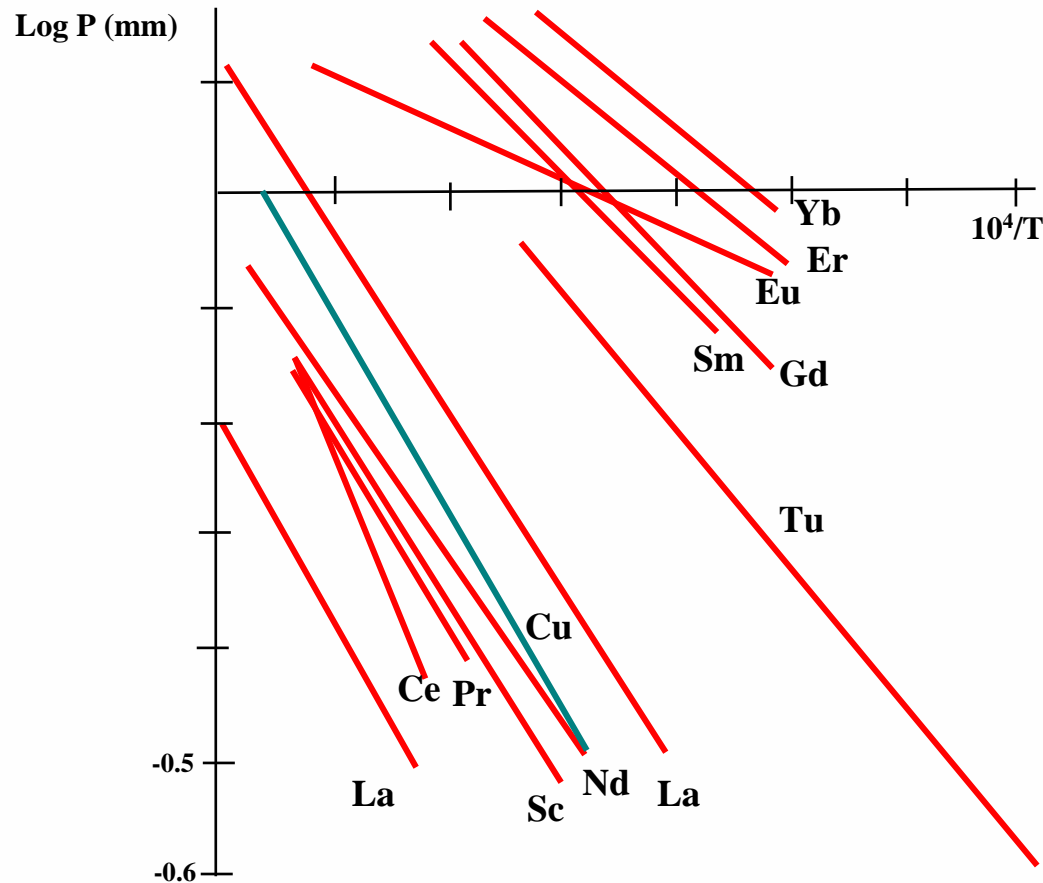
*Ref: 1995 Atomic Line Data (R.L. Kurucz and B. Bell) Kurucz CD-ROM No. 23. Cambridge, Mass.: Smithsonian Astrophysical Observatory.*

# Several Solutions for copper rate sensor

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- Chopped Ion Gage – gas pressure sensitive but robust.
- HCL lamp AA (Atomica<sup>TM</sup>) – saturation?
- Sum frequency generation and group velocity delay – shows promise.
- Nd/Cu congruent evaporation - sense Nd as entire R.E. or tracer.

# Copper and Neodymium Have Similar Vapor Pressures But close enough?



An. N. Nesmeyanov, Vapor Pressure of Elements, Academic, 1963, p150,p248



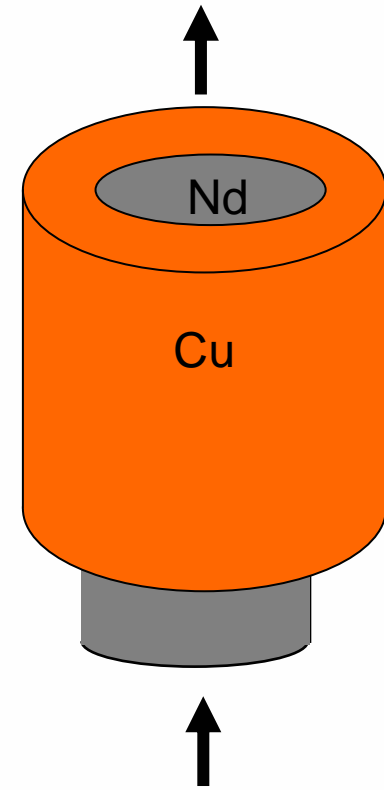
# Composition Results (ICP)

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	Nd	Ba	Cu	Cu/Nd	Note
Goal	17%	33%	50%	3	
First Run	2.73%	24.42%	72.86%	26.7	Graphite
Second Run	4.71%	24.10%	71.19%	15.1	Mo crucible
Third Run	15.58%	34.04%	54.38%	3.49	Mo crucible

# Solution – Rod Feed

- Rod is Cu-RE mixture
- The solid rod beneath the melt provides material for steady state evaporation with a melt pool of the correct composition for a stoichiometric vapor plume
- Has been used at industrial scales (300 kW).
- Ti-6Al-4V evaporation shows that it can be done with great stability.



# Summary

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- Co-evaporation is operational at LANL.
- Several copper sensors are being explored. We have full confidence in a solution.
- IBAD template, PLD buffers, PLD YBCO, GADDS XRD, state-of-art measurement on site.
- Stanford and LANL have an excellent collaboration.
  - Reliable data on temperature profile during batch process will be applied to LANL tape process.
  - Exchange of ideas is fruitful and ongoing.

# Performance FY 2004

## 1. Explorer the remained scale-up issues for CC & LANL

- More practical approach to Cu atomic absorption rate control – dope Cu with RE.  
(*Jonathan Storer LANL*)
- Explorer the Phase Formation – FTIR.
- Study Rate/Liquid Layer – X-ray DHS.

## 2. Apply Stanford High-R process on LANL IBAD tape

- Study the growth of YBCO films on tape with respect to the growth rate, deposition temperature, and post-deposition treatment.
- Characterized by XRD and R - T.
- Performance of our film in terms of  $J_c(\mathbf{B})$  (only measured on Xtl).
- $J_c$  measurement system construction (tape).



# Plan 2005

1. Continue Deposition of YBCO on IBAD Tape.
2. Make Optical Infrared Reflectance (FTIR) Available to Community as Desired.
3. Refine and Understand the Process on Tape
4. Modify the  $J_c$  &  $J_c(\mathbf{B})$  System for CC Tape Measurement.



# Results 2004

- *(Report by Jonathan Storer, LANL)*
  - More practical approach to Cu atomic absorption rate control – dope Cu with RE. Rate control on RE. Avoids problem of strong atomic absorption of Cu.
- *(Report by Hong-Ying Zhai, Stanford)*
  - Grew YBCO films on tape from 870° C – 940 ° C. Characterized by XRD and resistivity. Lower substrate temperature resulting improved performance.
  - Applied new FTIR characterization tool to tapes
    - Established directly temperature of film on tape during growth
    - Use FTIR reflectivity to monitor material state during deposition and processing.
  - Using X-ray Heating Stage, study the phase transitions in  $\text{YBa}_2\text{Cu}_3\text{O}_7$  and Ba-Cu-O system on both IBAD tape and Single Crystal.





# Research Integration

- With LANL IBAD Tape:  
survives 900 °C /  $2 \times 10^{-5}$  O<sub>2</sub> condition.
- YBCO films on IBAD Tape Were Analyzed in LANL.
- Try Establishing *in situ* Approach for LANL Facility.
- Collaboration with ORNL on J<sub>c</sub>(**B**) measurements.
- Applications of FTIR in the CC Community.
- Willing to Help to the Program With FTIR Tools.



# Team & Acknowledgments

## Team

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Gertjan Koster, Jung-hyuk Lee, Albert Chan,  
Arturas Vailionis, Hong-Ying Zhai

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# Thanks

